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ISO RECOMMENDATION

R 599

PLASTICS

DETERMINATION OF THE PERCENTAGE OF EXTRACTABLE MATERIALS IN POLYAMIDES

1st EDITION

August 1967

For each individual country the only valid standard is the national standard of that country.

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BRIEF HISTORY

The ISO Recommendation R599, *Plastics—Determination of the percentage of extractable materials in polyamides*, was drawn up by Technical Committee ISO/TC 61, *Plastics*, the Secretariat of which is held by the United States of America Standards Institute (USASI).

Work on this question by the Technical Committee began in 1958 and led, in 1961, to the adoption of a Draft ISO Recommendation.

This first Draft ISO Recommendation (No. 512) was circulated in June 1962 to all the ISO Member Bodies for enquiry. As the results of this consultation were not considered satisfactory, the Technical Committee presented a second Draft ISO Recommendation, which was circulated to all the Member Bodies in July 1964 and which was approved, subject to a few modifications of an editorial nature, by the following Member Bodies:

Austria	Italy	Sweden
Belgium	Japan	Switzerland
Canada	Korea, Rep. of	U.A.R.
Chile	Netherlands	United Kingdom
Czechoslovakia	New Zealand	U.S.A.
Denmark	Poland	U.S.S.R.
Finland	Romania	
Germany	South Africa, Rep. of	
India		

One Member Body opposed the approval of the Draft:

France

The second Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in August 1967, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 599 August 1967

PLASTICS

DETERMINATION OF THE PERCENTAGE OF EXTRACTABLE MATERIALS IN POLYAMIDES

1. SCOPE

1.1 This ISO Recommendation describes methods for the determination of the percentage of extractable materials in polyamides. These materials are for the most part composed of monomer and low molecular mass polymers, and water and possibly additives. Consequently, in order to know the percentage of monomer and low molecular mass polymers, it is necessary to determine the water content of the sample (see ISO Recommendation R . . . *). If the additive (for example, a plasticizer) is extractable by the liquid, the method cannot be applied without special procedure agreed between contracting parties.

1.2 Two methods are described:

— Method A, extraction with boiling water for polyamide 6 (polycaprolactam), and

— Method B, extraction with boiling ethanol for other polyamides (66, 610, 11, etc.).

1.3 The calculated percentage may be of importance when evaluating results found with the methods for

(*a*) determination of the viscosity number of polyamides in dilute solution (see ISO Recommendation R 307, *Plastics—Determination of the viscosity number of polyamides resins in dilute solution*), and

(*b*) determination of the " viscosity ratio " of polyamides in concentrated solution (see ISO Recommendation R 600 *, *Plastics—Determination of the viscosity ratio of polyamides in concentrated solution*).

NOTE.—It should be noted that the extract as determined by method A includes water where as the residue obtained by method B is the dry extract.

* At present Draft ISO Recommendation No. 1004, *Determination of the water content in polyamides.*

2. METHOD A FOR POLYAMIDE 6 OR ANY OTHER POLYAMIDE CONTAINING AMINOCAPROIC RESIDUE

2.1 Apparatus and supplies

(*a*) *Means* for reducing the sample to a finer state of division.

(*b*) *Extraction bag*, made from polyamide 66 thread (for example, 1.7 tex * woven to 20 threads per centimetre) with a diameter of about 7 cm and a height of about 18 cm. Before first use, the bag should be extracted in boiling distilled water for 24 hours.

(*c*) *Weighing bottle*, 100 ml with ground-glass stopper.

(*d*) *Flask*, 500 ml with ground neck.

(*e*) *Reflux condenser*, with ground joint to fit on the 500 ml flask.

(*f*) *Appropriate heating device* for the 500 ml flask.

(*g*) *Balance*, to weigh to 1 mg.

(*h*) *Sieve*, with mesh opening 2.36 mm.

(*i*) *Desiccator*, with calcium chloride.

(*j*) *Oven* maintained at 80 ± 2 °C.

(*k*) *Distilled water*.

2.2 Preparation of sample

Take a representative sample of the polymer and reduce it to small pieces to pass through the sieve (*h*), but not through the bag described under clause 2.1 (*b*). The water content should be determined previously.

2.3 Procedure

After extracting the bag 6 hours in boiling distilled water and drying at 80 ± 2 °C for 4 hours, weigh the bag in the weighing bottle to the nearest 1 mg. Introduce about 10 g of the sample with known water content into the bag and knot the ends of the bag. Weigh the bag with sample in the weighing bottle to the nearest 1 mg. Then immerse the bag with its contents in 250 ml distilled water in the 500 ml flask. Mount the reflux condenser on the flask and boil the contents of the flask for 6 hours. After pouring out the water, add a further quantity of distilled water and boil for another 10 minutes. Repeat this operation twice (three times in total). Then remove the bag from the flask, put again in the open weighing bottle and dry in an oven at 80 ± 2 °C for 12 hours. After cooling in the desiccator, stopper the weighing bottle containing the bag and weigh to the nearest 1 mg.

* The tex is the basic unit of the universal system for designating the count of textile fibres (see ISO Recommendation R 138, *Textiles—Universal Yarn Count System*). 1 tex=1 gramme per kilometre.

2.4 Calculation and expression of results

2.4.1 *The percentage of extractable materials* in the polyamide is calculated by the following formula:

$$\frac{P - P_1}{P} \times 100$$

where

P = mass of the sample before extraction, expressed in grammes,

P_1 = mass of the sample after extraction, expressed in grammes.

2.4.2 *The percentage of monomer and low molecular mass polymer content*, taking into account the water content of the sample, may be calculated by the following formula:

$$\frac{(P - U_1) - P_1}{P - U_1} \times 100$$

where

U_1 = water content of the sample, expressed in grammes,

$$U_1 = \frac{P \times U}{100}$$

P = mass of the sample before extraction, expressed in grammes,

U = percentage of water in the sample,

P_1 = mass of the sample after extraction, expressed in grammes.

2.4.3 Carry out two determinations. If they differ by more than 0.30% in absolute value, the test should be repeated. The average of two acceptable determinations is reported to the nearest 0.05%.

3. METHOD B FOR POLYAMIDES 66, 610, 11, ETC.

3.1 Apparatus and supplies

(*a*) *Means* for reducing the sample to a finer state of division.

(*b*) *Flask*, 300 ml with ground neck.

(*c*) *Reflux condenser*, with ground joint to fit on the 300 ml flask.

(*d*) *Water-bath.*

(*e*) *Oven*, maintained at 105 ± 5 °C.

(*f*) *Distillation apparatus*, consisting of a 300 ml distillation flask connected by a ground-glass joint to a condenser (for example, Claissen type apparatus with Liebig condenser).

(*g*) *Balance*, to weigh to 1 mg.

(*h*) *Hot filter funnel.*

(*i*) *Glass wool.*

(*j*) *Sieve*, with mesh opening 2.36 mm.

(*k*) *Desiccator*, with calcium chloride.

(*l*) *Ethanol* 95%, analytical grade.

3.2 Preparation of sample

Take a representative sample of the polymer and reduce it to small pieces to pass through the sieve (*j*). The water content should be determined previously.

3.3 Procedure

Weigh about 10 g polyamide to the nearest 1 mg and introduce it into the 300 ml flask with 150 ml ethanol. After mounting the reflux condenser, boil the content of the flask on the water bath for 6 hours. Filter the hot content of the flask through glass silk in the hot filter funnel into the distillation flask that is previously dried and weighed to the nearest 1 mg. Rinse the flask and the glass silk five times with 20 ml hot pure ethanol into the distillation flask. After mounting the condenser distil off all ethanol by heating the distillation flask on the water bath; the operation should be conducted carefully, without any entrainment of the extract. Place the distillation flask containing the residue in the oven, dry for 4 hours, and after cooling to room temperature in a desiccator weigh to the nearest 1 mg.

3.4 Calculation and expression of results

3.4.1 *The percentage of extractable materials* in polyamide is calculated by the following formula:

$$\frac{P_1}{P} \times 100$$

where

P = mass of the sample, expressed in grammes,

P_1 = mass of the residue, expressed in grammes.

3.4.2 *The percentage of monomer and low molecular mass polymer content*, taking into account the water content, is calculated by the following formula,

$$\frac{P_1}{P - U_1} \times 100$$

where

P = mass of the sample, expressed in grammes,

P_1 = mass of the residue, expressed in grammes,

U = percentage of water in the sample,

U_1 = water content of the sample, expressed in grammes,

$$U_1 = \frac{P \times U}{100}$$

3.4.3 Carry out two determinations. If they differ by more than 0.30 % in absolute value, the test should be repeated. The average of two acceptable determinations is reported to the nearest 0.05 %.